Quick and Simple Porosity Measurement at the Well Site

M.J. Dick¹, D. Green¹, T. Kenney¹, D. Veselinovic¹, J. Tallarita², and M.A. Smith²

¹Green Imaging Technologies, Fredericton, NB, Canada

²ALS Oil & Gas, Houston, TX, USA

This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Vienna, Austria, 28 August – 1 September 2017

Abstract

Porosity is the single most important petrophysical property. Typical measurements of porosity are done via down hole logging tools and core analysis. These methods either do not generate immediate porosity data while drilling, are complex, expensive, or prone to error. Furthermore, modern drilling produces cuttings that are not suitable for most conventional porosity measurements as they are crushed into very small "grain like" pieces. Nuclear Magnetic Resonance (NMR) measurements overcome the shortcomings of traditional porosity measurements allowing porosity to be determined efficiently and accurately on drill cuttings. In addition, the NMR measurement of the cuttings provides not only the porosity but the pore size distribution as well (assuming the cuttings are not crushed beyond the pore size).

In this work, we propose a simple measurement of porosity using benchtop NMR on modern drill cuttings. The NMR technique has been tested on both shale and sandstone samples to date. For the shale samples, T₂ distributions were used to determine the porosity of core plugs, crushed core plugs and drill cuttings. The results showed excellent agreement between porosities derived for each sample. For the sandstone samples, T₂ distributions were used to determine the porosity of core plugs and drill cuttings which were saturated with water in the lab. The results showed good agreement between the porosity derived for each sample. Some error was observed due to extra water present on the surface of the cuttings. We have completed extensive work in optimizing our experimental technique to minimize this error. This refinement of our experimental technique will be described in this paper. Currently we are furthering our testing by using samples from different fields predominately from unconventional reservoirs.

Introduction

Knowing the porosity of the rocks in an oil field is vital to the profitable development of the field. The porosity is reflective of the amount of oil present in a field. The earlier porosity is known the earlier decisions can be made about how to best retrieve the oil from the field. Typical measurements of porosity are done via down hole logging tools and core analysis. These methods either do not give immediate feedback of the porosity while drilling, are complex, expensive, or prone to error. Nuclear Magnetic Resonance (NMR) is widely used in the oil and gas sector to investigate both the types of fluids present and the porosity of the oil bearing rocks [1]. NMR measurements are well suited to overcome the shortcomings of traditional porosity measurements allowing porosity to be determined efficiently and accurately on drill cuttings.

SCA2017-013 2/10

NMR measurement of the cuttings provides not only the porosity but also the pore size distribution (provided the cuttings are not crushed beyond the pore size). Ignoring diffusion, the relationship between the NMR property T_2 and the pore size is governed by the following equation.

$$\frac{1}{\mathsf{T}_2} = \frac{1}{\mathsf{T}_{2-\mathsf{Bulk}}} + \rho \frac{\mathsf{S}}{\mathsf{V}} \tag{1}$$

Where S/V is the surface to volume ratio of the pore, ρ is the relaxivity parameter and T₂-bulk is the T₂ relaxation time of the fluid. The surface, S, to volume, V, ratio is the pore size and if the other terms are ignored, is directly related to the T₂ through the relaxivity parameter, ρ . Therefore, a plot of volume (retrieved from NMR signal) vs T₂ is the pore size distribution. The pore size distribution offers complimentary information to porosity i.e. what size pores the oil is in and hence how hard it will be to retrieve. The ability of NMR measurements to determine the pore size distribution of drill cuttings makes it superior to traditional measurements done on cuttings which don't provide the pore size distribution. In this paper, we summarize work that has been completed on developing an effective technique to measure the porosity and pore size distributions on drill cuttings.

Experiment

In the initial tests, T₂ NMR acquisition scans for the dry or as received sandstone and shale plugs, crushed plugs and cuttings were recorded using an Oxford Instruments GeoSpec 2-53 rock core analyzer [2]. Data acquisition and data analysis of the T₂ data was achieved via Green Imaging Technology software [3]. Comparison between the T₂ data for the plugs, crushed plugs and cuttings validated the ability of NMR to accurately measure the pore size distributions of drill cuttings.

Next the ability of NMR to accurately determine the porosity of cuttings was tested by, again, comparing data derived from plugs, crushed plugs and cuttings. The porosity of the cuttings and crushed plug were determined using the following procedure.

- 1. Run an NMR T_2 scan on a vial filled to a pre-determined level with the saturating fluid to measure the total volume, V_{total} .
- 2. All the samples were vacuum saturated with brine (2% KCl in water) for approximately an hour.
- 3. The samples were then removed from the brine and their pore volumes and T_2 distributions were retrieved.

For the core plugs this is the end of the procedure. The pore volume retrieved along with the bulk volume is used to determine the porosity of the plug. For the cuttings and crushed core plugs, pore volumes retrieved from the NMR data corresponded to the volume of the cuttings (V_{cuttings}). This data was used in the remainder of the procedure to determine the porosity of the cuttings and crushed plugs. In order to capture any possible residual bulk water signal, the following NMR parameters were employed; Tau was set at $100\mu s$, recycle delay was 28,125ms and T_2 max was 3,750ms.

SCA2017-013 3/10

4. Place the saturated cuttings or crushed core plug into the vial from Step #1 and remove any fluid above the pre-determined level. Run a T_2 scan to measure the volume of the vial's contents, $V_{\text{cuttings+fluid}}$.

5. Calculate the porosity of the cuttings using the equation:

$$Porosity = \frac{Pore \ volume \ of \ cuttings}{Bulk \ volume \ of \ cuttings} = \frac{V_{cuttings}}{V_{total} V_{cuttings+fluid} + V_{cuttings}}$$
(2)

This procedure is straightforward and easy to implement. However, it was found to overestimate the porosity of the cuttings and crushed plugs due water stuck to the surface of the cuttings. This water has NMR signal and as a result V_{cuttings} is often inflated in the NMR data leading to a higher than expected porosity value. The majority of the work summarized in this paper has centered on deriving an optimal technique for removing this surface water without compromising the water in the pores of the cuttings. These efforts will be described in the following section.

Results

Pore Size Distribution Of As Received Samples:

Figure 1 shows a comparison of the pore size distribution for a core plug (green trace), a crushed core plug (blue trace) and cuttings (red trace). All samples came from the same shale formation and were tested in their as received or dry state. Each sample had the same pore size distribution with a peak near a relaxation time of 0.2 ms. This relaxation time is typical of shale samples. The excellent agreement between the pore size distribution of each sample indicated that pore size distribution and hence the porosity of samples is invariant from core plug to cuttings. Several shale samples were tested in this manner and all showed good agreement of their pore size distributions for core plugs, crushed core plugs and cuttings.

The pore size distribution for core plugs, crushed core plugs and cuttings of several sandstone samples were also compared. They also showed good agreement of the retrieved pore size distributions. The only difference between shale and sandstone was that if the sandstone cuttings were crushed to a size smaller than the typical pore size then the pore size information derived from the cuttings would be compromised. This is not the case for shales which have very small pores making it unlikely that the cuttings would ever be crushed smaller than the typical pore size.

Pore Volume Retrieval Of Saturated Sandstone Samples:

Figure 2 compares the pore size distribution for a plug (blue trace) and a crushed plug (red trace) taken from the same sandstone formation. The porosity of the crushed plug was determined using the procedure outlined in the last section. The area under each curve is the cumulative porosity for each sample. If all else is equal, the cumulative porosity for each sample should be very similar as the samples were derived from the same formation. From the figure, it is clear that the porosity derived from the crushed core plug is higher

SCA2017-013 4/10

than that derived from the core plug. This is due to extra NMR signal from water stuck to the surface of the crushed samples.

This becomes more obvious when the pore size distributions in Figure 3 are examined. In this case, the water on the exterior of the crushed plug has a longer T₂ relaxation time than the water in the pores. This results in a shoulder present on the peak of the porosity distribution for the crushed core plug (red trace) and yields a higher cumulative porosity as compared to the core plug (blue trace). Table 1 summarizes the difference between porosities derived from NMR data for core plugs vs. crushed core plugs for several sandstone samples tested. In each case the porosity derived from the crushed plug overestimates the porosity by between 2.4 and 6.8 porosity units.

Pore Volume Retrieval Of Drill Cuttings:

The overarching goal of this work has been to determine a quick, efficient and effective method of determining the porosity characteristics of cuttings such that this process can be performed on-site, while drilling. A set of cuttings was provided to us by ALS from one of their drill sites for refinement of our procedure. This was meant to be a blind study and ALS provided us no information on the type rock the cuttings originated from. The conventional method of removing excess liquid by means of a dampened paper towel (API 5.2.4.6.2 [4]) was not viable in this case due to the delicate and fragile nature of the cuttings in question. So instead, the procedure outlined in the Experimental section was modified as follows; steps one and two were followed as described, step three was skipped and finally step four was also followed as described. Once V_{cuttings+fluid} was determined, the brine solution was displaced from around the ALS cuttings by means of introducing a more dense, NMR invisible, fluid, namely fluorinert (FC-40). After the FC-40 was introduced the water, now floating on top of the emulsion, was siphoned off by pipette; at this stage V_{cuttings} could be established by performing a T₂ NMR scan.

However, as with the original procedure, the $V_{cuttings}$ T_2 NMR scan provided an erroneously high volume value for the cuttings; giving porosity values of 55% and higher, which exceeds the generally accepted sandstone porosity range of 10-40% [5], to say nothing of shale. It was hypothesized at this point, that the spurious signal seen in the $V_{cuttings}$ scans was due to contamination of the cuttings caused by the drilling mud ALS used for boring; thus, ALS was consulted and new cuttings, cleaned per their standard Soxhlet based procedure, were provided. However, after repeating the above described procedure to derive V_{total} , $V_{cuttings+fluid}$ and $V_{cuttings}$, the average porosity of the three retested samples was still 54%. Due to there being no significant impact on the calculated porosity figures after cleaning the cuttings it was then hypothesized that the FC-40 was not displacing all of the brine. As a result, the new experimental procedure was still subject to surface water contamination and hence artificially inflating the $V_{cuttings}$ value, leading to an overestimation of the cuttings' true porosity.

Development Of A Technique To Remove Surface Water From Crushed Samples:

SCA2017-013 5/10

As a result of the failure of our experimental procedures to accurately predict the porosity of both crushed core plugs and drill cuttings, it was decided that a more controlled development of a technique to remove surface water was needed. A sandstone plug with known parameters, NMR peak, pore volume and porosity, was selected. A slice of this plug, approximately 0.5cm in length, was removed from the main body. Both the plug and the slice of the plug were then immersed in methanol and cleaned using the Dean-Stark cleaning procedure. After the methanol cleaning, the plug and slice were transferred to toluene for removal of possible oil content, again, via the Dean-Stark cleaning procedure. After cleaning was completed, the slice was pulverized into pieces 1 to 3 mm in diameter. The porosity of the plug was then determined using the pore volume measured by NMR and the bulk volume. This porosity would act as the standard all further tests on the pulverized pieces would be compared against.

The initial test on the crushed plug was to employ the procedure outlined in the last section on the crushed pieces. The left most bar in Figure 4 shows the percent difference between the porosity retrieved from the crushed plug with the porosity of the plug itself. The 97% difference between the two porosities was a clear indication that the technique of FC-40 displacement of surface water did not work effectively.

To further understand how to best eliminate surface water from the crushed plug pieces, six experimental assay groups were tested. They are as follows; screening or size differentiation, D₂O washing, variable centrifugal speed, surfactant infused fluorinert, CuSO₄ doped H₂O and sonication. These methods were devised to eliminate the persistent surface or bulk water signal in the V_{cuttings} derived from NMR analysis. For each assay, the original procedure outlined above was followed to generate, clean and saturate the crushed plug pieces. A comparison of the porosity derived from each procedure with the known porosity of the standard core plug is shown in Figure 4.

The screening assay underwent the same procedure as the original cuttings, however, immediately after pulverization of the plug slice, the cuttings were poured over a wire screen with openings of 1.45 mm. This yielded a percent difference in the calculated porosity of 90%.

The D₂O washing assay followed the original procedure to generate, clean and saturate the crushed plug pieces. The difference in the D₂O washing assay arose after the V_{brine+cuttings} scan was completed. The brine surrounding the cuttings was removed, as much as possible, by pipette. Afterwards, instead of immediately introducing FC-40 to displace the brine, 5ml of D₂O was first mixed in with the remaining brine. It was thought that the D₂O would mix with any remaining surface water on the crushed core pieces so that any water that remained bound to the cuttings by surface tension would be NMR invisible. After the D₂O was mixed with the brine, FC-40 was introduced to displace and remove the D₂O to prevent it from infiltrating the pore spaces of the cuttings. A variation on this method, D₂O rinsing, simply used the 1.45mm screen to hold the cuttings while 5ml of D₂O was poured over them instead of introducing the D₂O directly into the vial. According to Figure 4, the percent difference for these two methods were 38% for the D₂O washing and 20% for the D₂O rinsing.

SCA2017-013 6/10

The surfactant assay followed the original procedure but the FC-40 was mixed with a surfactant, SDS, before being introduced to the vial. It was thought that this might aid in the mixing of the existing brine and FC-40 such that it would encourage the removal of the cuttings' surface water. This yielded a percent difference in the calculated porosity of 96%.

The CuSO₄ doped H₂O assay was identical in procedure to the original method but, before the introduction of FC-40, the brine present in the vial was removed via pipette and doped H₂O was then mixed in with the remaining brine in the vial. It was hoped that the doped H₂O would mix enough with the surface bound water on the cuttings such that it would separate the surface water signal from that of the water in the pores of the crushed pieces, at which point it could be easily and definitively subtracted. The concentration of CuSO₄ used was approximately 260g/L. This yielded a percent difference in the calculated porosity of 76%.

The next tested method was sonication. A Branson 3800 sonicator was filled with water and used to vigorously vibrate a partially immersed vial with the crushed core pieces and FC-40 in it. It was thought that such high frequency vibrations would remove any brine still clinging to the outside of the cuttings; the sonication lasted a total of 30 minutes. This yielded a percent difference in the calculated porosity of 64%.

The final methods tested involved the use of a centrifuge. The variable centrifuge speed method followed the original method. However, after the introduction of FC-40 to the vial, the immersed samples were spun at a range of centrifuge speeds. This centrifugal assay began at 100 RPM and increased in speed up to 7500 RPM with T₂ NMR scans (V_{cuttings}) being taken after each speed, each speed step lasted for 15 minutes. Figure 5 shows the detected volume at each centrifuge speed. From the figure, a clear drop in the detected volume is seen between 500 and 600 rpm and between 1000 and 1100 rpm. The T₂ NMR scans taken at these speeds show significant reduction to the long T₂ components. We interpret this reduction to be water leaving the surface of the crushed pieces. If the V_{cuttings} is taken as the detected volume at 1100 rpm then the percent difference is 9.4% between the calculated porosity and known porosity. We interpret any further decrease in detected volume beyond 1100 rpm to be due to water leaving the pore volume due to the everincreasing centrifugal force. This is further confirmed by the T₂ NMR scans at these speeds which show any further reduction in detected volume coming from the shorter T₂ components corresponding to water in the pores.

Another experimental assay involving centrifugation was time varied centrifugation. Based on the results from the variable centrifuge speed method, 1100 rpm was selected as the centrifuge speed which captured most of the crushed pieces surface water loss. The pieces were then spun at 1100 rpm for varying amounts of time. The detected volume was then measured via T₂ NMR scans after each spin time. After 36 minutes of centrifugation the detected volume yielded a porosity within 4 % of the expected porosity.

To determine the repeatability of the centrifugation technique, four samples of crushed pieces were processed using centrifugation at 1100 rpm for 35 mins. The retrieved volumes after centrifugation all yielded porosities within 3% of the expected porosity.

SCA2017-013 7/10

Testing of the centrifugation technique using ALS cuttings

Once a reliable method for determining the porosity of crushed pieces was determined, our investigation turned back to the drill cuttings provided by ALS Oil & Gas. These cuttings were processed using the centrifugation technique described in the last section with centrifugation taking place for 35 min at 1100 rpm. Unfortunately, the porosities retrieved after centrifugation were still higher than expected for a reservoir rock. However, the T2 spectra recorded after centrifugation showed no sign of surface water indicating that the extra porosity observed is of another origin. As mentioned above, we have investigated the possibility that the cuttings have been contaminated by drilling mud yielding higher than expected porosities. Cleaning and drying the cuttings has not yet yielded a reasonable porosity. Another possibility is that the cuttings have been crushed beyond the assumed pore size of the rocks (the actual pore size for these samples is not known) rendering NMR analysis ineffective. Investigations continue into retrieving an accurate porosity of the ALS cuttings.

Conclusion

A method has been presented for using NMR to determine the porosity of crushed rock samples. An intensive investigation has gone into first showing that crushing or cutting rock samples does not compromise their pore networks. As a result, NMR remains a feasible way of determining the porosity of crushed rock samples. Next an extensive investigation into determining the best method to accurately determine the porosity of crushed samples has been carried out. It was discovered that during water saturation of the crushed samples, their surfaces become contaminated with water. Removing this water without removing water in the sample's pore network has proven to be difficult. A procedure involving centrifuging the samples was developed and seems to be effective in eliminating the unwanted surface water without eliminating the water in the pores. When the centrifugation procedure was carried out on drill cuttings provided by ALS Oil & Gas, the porosities derived were too high. Work continues on determining why our experimental technique was not effective on the ALS cuttings.

References

- 1. Coates, G.R., Xiao, L., and Prammer, M.G., *NMR Logging. Principles & Applications*, Halliburton Energy Services, Houston, 1999.
- 2. Geo-Spec 2-53 User Manual, Version 1.8, Oxford Instruments.
- 3. GIT Systems and LithoMetrix User Manual, Revision 1.9, Green Imaging Technologies.
- 4. American Petroleum Institute, *Recommended Practices for Core Analysis*, API Publications, Washington, D.C., (1998), p. 5-7.
- 5. Pittman, E. D., *Porosity diagenesis and productive capability of sandstone reservoirs*, (1979).

SCA2017-013 8/10

Tables and Figures:

Sandstone	A	В	С	D
Core Plug	16.7	14.1	16.0	15.2
Crushed Core Plug	23.2	16.3	18.4	22.0

Table 1: NMR porosities for core plugs vs. crushed core plugs

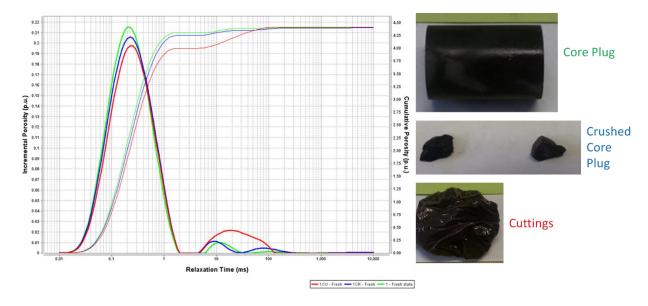


Figure 1: Comparison of the pore size distribution for a shale core plug (green trace), a crushed core plug (blue trace) and cuttings (red trace).

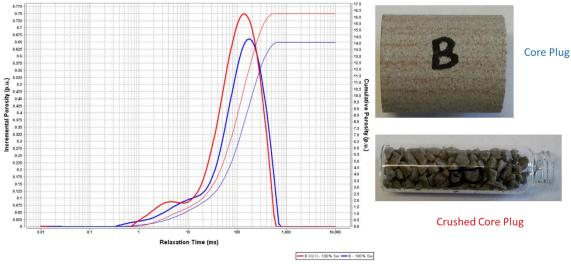


Figure 2: Comparison of the pore size distribution for a sandstone core plug (blue trace) and a crushed core plug (red trace).

SCA2017-013 9/10

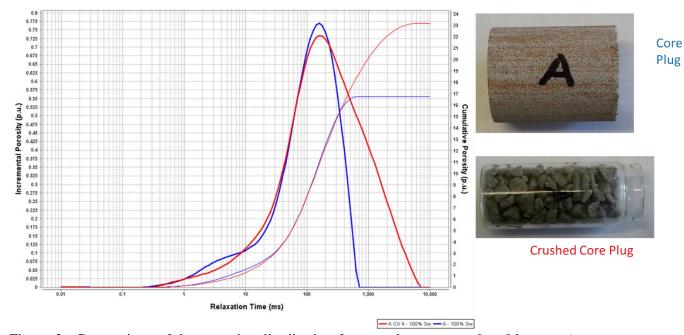


Figure 3: Comparison of the pore size distribution for a sandstone core plug (blue trace) and a crushed core plug (red trace).

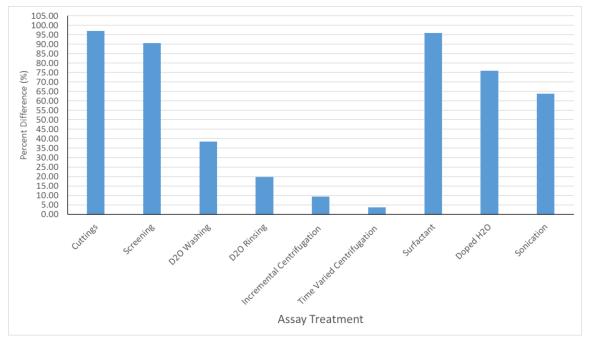


Figure 4: Bar graph comparison of the porosity determined for each procedure derived to eliminate the persistent surface water with the known porosity of the standard core plug

SCA2017-013 10/10

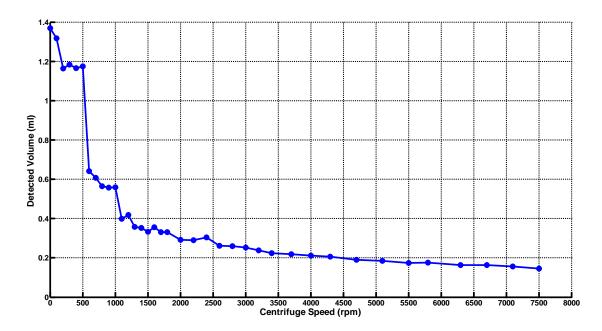


Figure 5: Detected volume in crushed sandstone pieces as a function of centrifuge speed